

INSIGHTS ON STATE-OF-THE-ART QUALITY CONTROL

Learn how to use the latest materialographic preparation techniques to examine plasma spray coatings to ensure the coatings have the required integrity for the application.

By Judith Arner
Senior Metallographer, Struers



PLASMA SPRAY COATINGS

PREFACE

Quality assurance is a critical process in many industries. Quality control ensures customers receive products that meet their needs and are free from defects.

Manufacturers are under constant pressure to meet market demands for safety, operational stability, and environmental standards. At the same time, they are constantly looking to improve productivity and reduce costs in order to remain competitive in a global market.

This preparation report shows you how to use a state-of-the-art metallographic preparation process to reveal the true structure of an applied coating effectively – so you can check for quality.



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THE FIELD OF STUDY

Although tremendous advances have been made in coatings in recent years, many companies lag behind in terms of the methods they use for metallographic preparation of applied coatings. As new alloys for coatings are being developed, it is more important than ever that you understand how sample preparation can affect how a coating is evaluated.

Why materialography?

Regardless of how you use a coating, its engineered surface needs to be analyzed for:

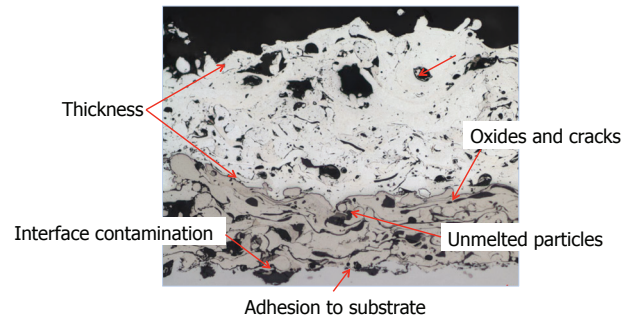
- Quality control
- Monitoring application processes
- Failure analysis

Materialography also helps in the development of new products and the development of new application methods.

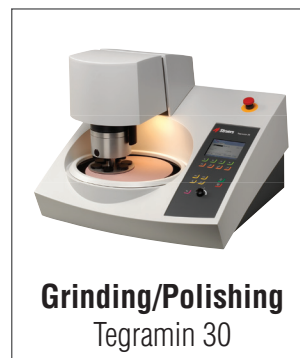
Proper metallographic preparation is important as it enables you to examine the following properties of a coating, which are all key parameters in quality control:

- Thickness
- Interface contamination
- Adhesion to substrate
- Unmelted particles
- Oxides and cracks
- Porosity

The objective of this study was to apply a reproducible preparation method – including cutting, mounting, grinding and polishing – that would reveal key parameters without introducing artifacts, such as cracks, reliefs, smearing or pull outs.



Solution



Cutting:



- Equipment: Discotom-100

Challenge: To clamp the substrate gently and select the appropriate cutting parameters to ensure that the adhesion between the substrate and coating is maintained.

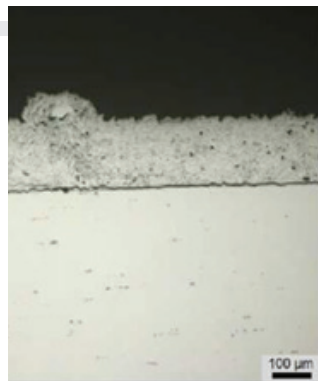
Method: A coating is typically a fraction of the substrate's thickness. Therefore, you should select a cut-off wheel to best suit the substrate. The coating should be protected with padding so that the clamp is not in direct contact with it.

For fragile coatings, apply an epoxy mount before cutting.

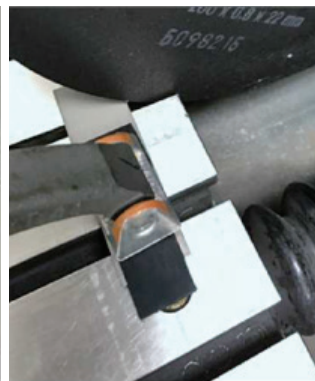
When delamination occurs, it is impossible to say whether it was due to the application process or sectioning. However, if epoxy is present in the gap (see image 4), delamination clearly occurred prior to mounting. If you are in doubt, section and mount another sample while paying very close attention to your methodology.



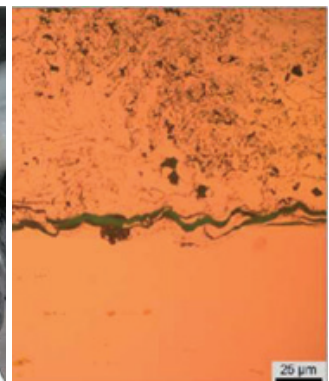
1 No delamination after cutting



2 Delamination after cutting



3 Protecting with padding



4 Delamination after cutting enhanced with Epodye

Mounting



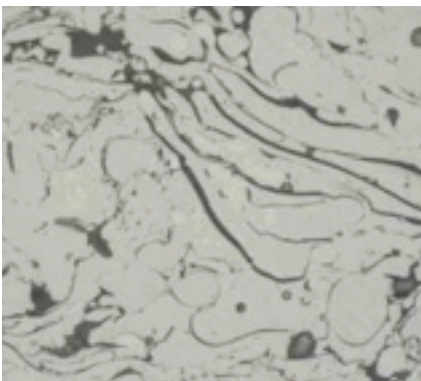
- Equipment: CitoVac
- Resin: Epofix
- Size of mounts: 30mm

To impregnate all of a sample's voids and pores and encapsulate brittle, porous coatings in epoxy is a challenge.

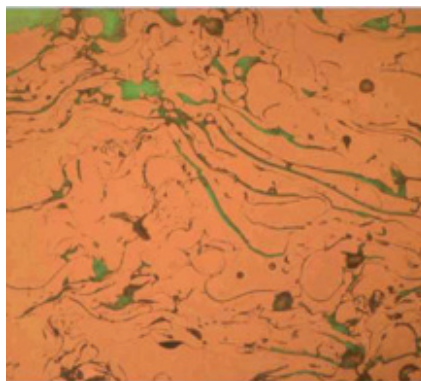
Vacuum impregnation can be used to overcome this difficulty if the coating has a high degree of porosity. Vacuum impregnation can only be done with 'cold mounting' epoxy, as opposed to acrylics or hot compression mounting. The depth of impregnation depends on the degree of porosity and the interconnection between pores.

Hard, dense coatings should not be hot mounted. This is because these coatings are brittle and can be badly damaged by hot mounting. Instead, encapsulate brittle and porous coatings in epoxy.

If a coating has porosity or voids, use a resin dye during the epoxy vacuum impregnation. This helps identify pores, voids and oxides, as these appear dark under a microscope. All pores and voids should be filled with dyed epoxy prior to preparation so that they have a different appearance to the dark oxides in the coating.



1 No fluorescence



2 With fluorescence



3 Set up in the vacuum chamber

Grinding/Polishing



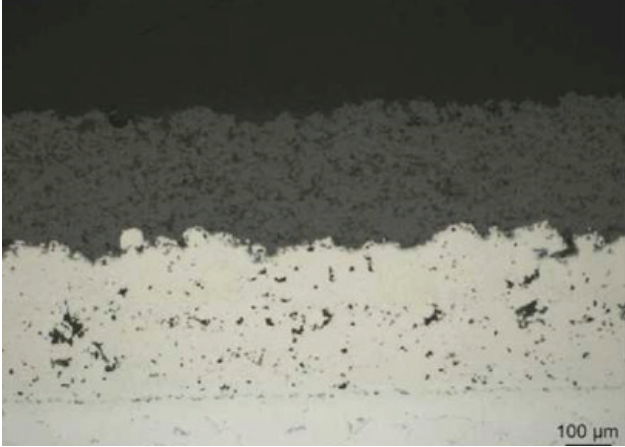
- Equipment: Tegramin 30
- Disc diameter: 300mm (12")
- Specimen holder: 02606916 – MAXCY

When selecting the ideal grinding/polishing method, you should take the following factors into account:

- Plane grinding must remove material without causing further damage. If done properly, polishing times are minimized.
- Diamond polishing is required to produce a flat surface free of deformations that can be viewed under a microscope.
- Oxide polishing is the final step in creating the best possible polished surface, one that enables you to prepare micrographs for reports.
- Moderate to low resilience polishing cloths minimize the rounding of voids and edges.
- Extensive polishing to reveal porosity should be avoided as this causes flatness to be lost, which can cause problems in measuring coating thickness.

Step	1	2	3	4
⊙ Disc / Cloth	SiC Foil #220	MD-Largo	MD-Dac	MD-Nap
Speed [rpm]	300	150	150	150
☼ Suspension		DiaPro Allegro/Largo 9µm	DiaPro Dac 3µm	DiaPro Nap B 1µm
Pre-dosing		3	3	3
Dosing		6	10	10
☼ Lubricant	Water			
Pre-dosing				
Dosing				
(F) Force [N] Single/Holder	30/180	30/180	30/180	20/120
Holder speed	150	150	150	150
↻ Rotation direction	>>	>>	>>	>>
⌚ Time [min]	1:00	5:00	5:00	1:00
Pause between steps	No	No	No	No

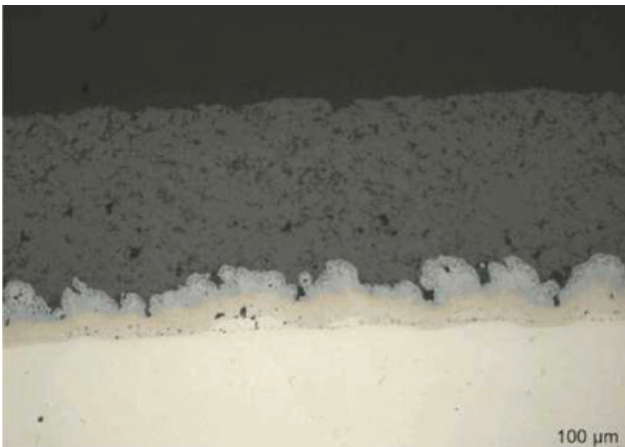
P 2 Step: MD-Nap with DiaPro Nap B can be replaced by MD-Chem with OP-U for metal coatings or OP-S for ceramic coatings.



1 Air Plasma Spray (APS) ceramic, aluminide bond coat



2 Plasma-sprayed molybdenum coating



3 Thermal barrier coating (TBC), aluminide bond coat

RESULT AND OVERVIEW

This application report gives you a reproducible method for the metallographic preparation of spray-coated samples. The method consists of:

- Cutting a coating sample while maintaining adhesion between the substrate and the coating.
- Cold mounting in epoxy under vacuum to impregnate all the voids and pores in a sample and encapsulate brittle, porous coatings in epoxy.
- Plane grinding with SiC to remove any cracks caused by cutting without introducing new damage from coarse grinding.
- Fine grinding on a rigid disc to produce a flat sample.
- Diamond polishing on a cloth to retain a sample's flatness and ensure the removal of smeared metal.
- Oxide polishing to create high structural contrast to make evaluation easier.

Solution



Cutting
Discotom-100



Mounting
CitoVac



Grinding/Polishing
Tegramin 30

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